# THE EFFECTS OF SINTERING PARAMETERS AND PARTICLE SIZE DISTRIBUTIONS ON P/M STEEL FORGINGS

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and

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U.S. ARMY ARMAMENT COMMAND

## RESEARCH DIRECTORATE

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The effects of sintering parameters and particle size distributions on the properties of steel P/M forgings were investigated. Sintering parameters studied were hydrogen flow rate, methane flow rate, and sintering time and temperature. Various unimodal and bimodal particle size distribututions designed from statistically determined powder blends were investigated in the powder distribution study. Oxygen content and final density in the forgings strongly depend on the proper selection of the sintering parameters. Among the sintering parameters studied, hydrogen flow rate (Cont) OVER

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20. Abstract (cont)

and temperature predominately influence the final P/M steel forging properties. The tensile and yield strengths of the P/M forgings were not sensitive to particle size distribution differences. Ductility and impact properties, however, improve with small particle size distributions. The increase in the ductility and impact properties correlates with lower oxide content, which in turn, is attributed to the decrease in oxide inclusions present in the powder particles. (Crowson, A. and Anderson, F. E.)

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## INTRODUCTION

Powder metallurgy (P/M) steel forging has received increased attention during the past few years.[1,2,3] As a viable production technique, cost advantages can be realized through more efficient material utilization and reduction of machining operations. Depending on the application, steel powders can be forged cold (less than 500°F)[4,5] or hot (1500 to 2200°F).[6,7] The basic process involves five steps:

Powder → Preform → Sinter → Forge → Finish

Each of these steps requires considerations peculiar to the P/M forging process to obtain good quality forgings.

The primary thrust of P/M steel forging development has been to reproduce the properties of conventionally forged products in the P/M forged part.[8,9] Attainment of these properties requires closing voids normally present in standard P/M parts in such a manner as to fully densify and achieve a sound metallurgical bond during forging.[10] Significant lateral

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1 "Forging of P/M Preforms", Modern Developments in Powder Metallurgy, (Ed. H.H. Hausner), Plenum Press, NY, 1971, Vol. 4, p. 369-523.

2 R.F. Halter, "Recent Advances in the Hot Forming of P/M Preforms", Modern Developments in Powder Metallurgy, (Ed. H.H. Hausner), Plenum Press, NY, 1973, Vol. 7, p. 137-152.

3 R. Gold, "Production P/M Hot Forging is Here", Precision Metal, Vol. 33, No. 11, 1975, p. 23-26.

4 Gustafen, D.A., "HD: P/M = High Density Parts via P/M Techniques", Metal Progress, Vol. 101, No. 4, 1972, p. 49-58.

5 R.H. Hoefs, "High-Density Cold-Pressed Parts Substitute for Ferrous Castings", Metal Progress, Vol. 107, No. 2, 1975, p. 71-80.

6 H.F. Fischmeister, L. Olsson, and K.E. Easterling, "Powder Forging", Powder Metallurgy International, Vol. 6, No. 1, 1974, p. 30-39.

7 Lally, F.T. and Toth, I.J., "Forged Metal Powder Gears", Technical Report No. 11960, September 1974.

8 Cull, G.W., "Mechanical and Metallurgical Properties of Powder Forgings", Powder Metallurgy, Vol. 13, No. 26, 1970, p. 156-164.

9 Lally, F.T., Toth, I.J., and DiBenedetto, J., "Forged Metal Powder Products", SWERR-TR-72-51, August 1972.

10 M.J. Koczak, C.L. Downey, and H.A. Kuhn, "Structure/Property Correlations of Aluminum and Nickel Steel Preform Forgings", Powder Metallurgy International, Vol. 6, No. 1, 1974, p. 13-16.

deformation under high forging temperatures and pressures is required to achieve this effect. As a result, the processing route (powder characteristics, preform density, lubrication, sintering conditions, and forging parameters) significantly affect the structural integrity and the attendant mechanical properties of the P/M forging. Consequently, careful control of the processing route is adamant to insure reproducible properties equivalent to those of conventional forgings of similar composition.

Because of the importance of the processing route, studies involving investigations into the effects of processing variables on final forged P/M steel properties have been initiated at Rock Island Arsenal. Previous efforts concentrated on the variables of preform density, lubrication, deformation, forging temperature, and forging pressure.[11] The purpose of the following study was to investigate the effects of sintering conditions and particle size distributions on the physical and mechanical properties of steel P/M forgings.

The study was divided into two sections: the first established the effects of sintering conditions, and the second determined the effects of particle size distributions on the steel P/M forged properties. The sintering parameters studied were flow rate of reducing gas, flow rate of carburizing gas, sintering time, and sintering temperature. Various unimodal and bimodal distributions designed from statistical powder blends were used in the particle size distribution study.

## SINTERING STUDIES

### Procedure

Commercially available prealloyed 4600 steel powder was used in the sintering studies. The chemical analysis of the powder is shown in Table 1 along with the chemical analysis required by AISI specifications for wrought 4600 metal. The 4600 steel powder had a composition which was modified (lower manganese, higher molybdenum) ostensibly to promote processing characteristics. Flake graphite was added to the 4600 powder to obtain 0.4-percent carbon content. The powder was characterized according to size and size distributuion (ASTM B-214-56), apparent density (ASTM B-212-48), and flow rate (ASTM B-213-48) as shown in Table 2.

Individual particles were evaluated by scanning electron microscopy (SEM) and standard optical metallography; see Figure 1. SEM examinations showed a distribution of sheroidal to popcorn-like shapes. Metallographic examinations showed the microstructure of the 4600 powder particles to have a generally uniform grain size of ASTM No. 12.

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11 A. Crowson, R.J. Grandzol, and F.E. Anderson, "Properties of P/M Steel Forgings", presented at Powder Metallurgy in Ordnance Up-Date Seminar, Metal Powder Industries Federation, 1975, in press.

TABLE I

CHEMICAL ANALYSIS OF 4600 PREALLOYED POWDER

| Element | AISI Specification | A.O. Smith EMP 4600 |
|---|---|---|
| Carbon | ----- | ---- |
| Nickel | 1.65 - 2.00 | 1.77 |
| Molybdenum | 0.2 - 0.3 | 0.48 |
| Manganese | 0.6 - 0.8 | 0.23 |
| Copper | ----- | 0.05 |
| Chromium | ----- | 0.05 |
| Phosphorus | 0.04 max | <0.01 |
| Sulfur | 0.04 max | 0.02 |
| Silicon | 0.02 - 0.34 | 0.07 |
| Oxygen | ----- | 0.152 |

TABLE 2

CHARACTERISTICS OF 4600 PREALLOYED POWDER

| U.S. Screen Size | Sieve Analysis, wt% |
|---|---|
| +80 (180μ) | 0.1 |
| -80 +100 | 4.5 |
| -100 +140 | 14.5 |
| -140 +200 | 23.4 |
| -200 +230 | 7.5 |
| -230 +325 | 18.7 |
| -325 (44μ) | 31.3 |
| Apparent Density ($g/cm^3$) | 2.99 |
| Flow Rate (sec) | 25.8 |

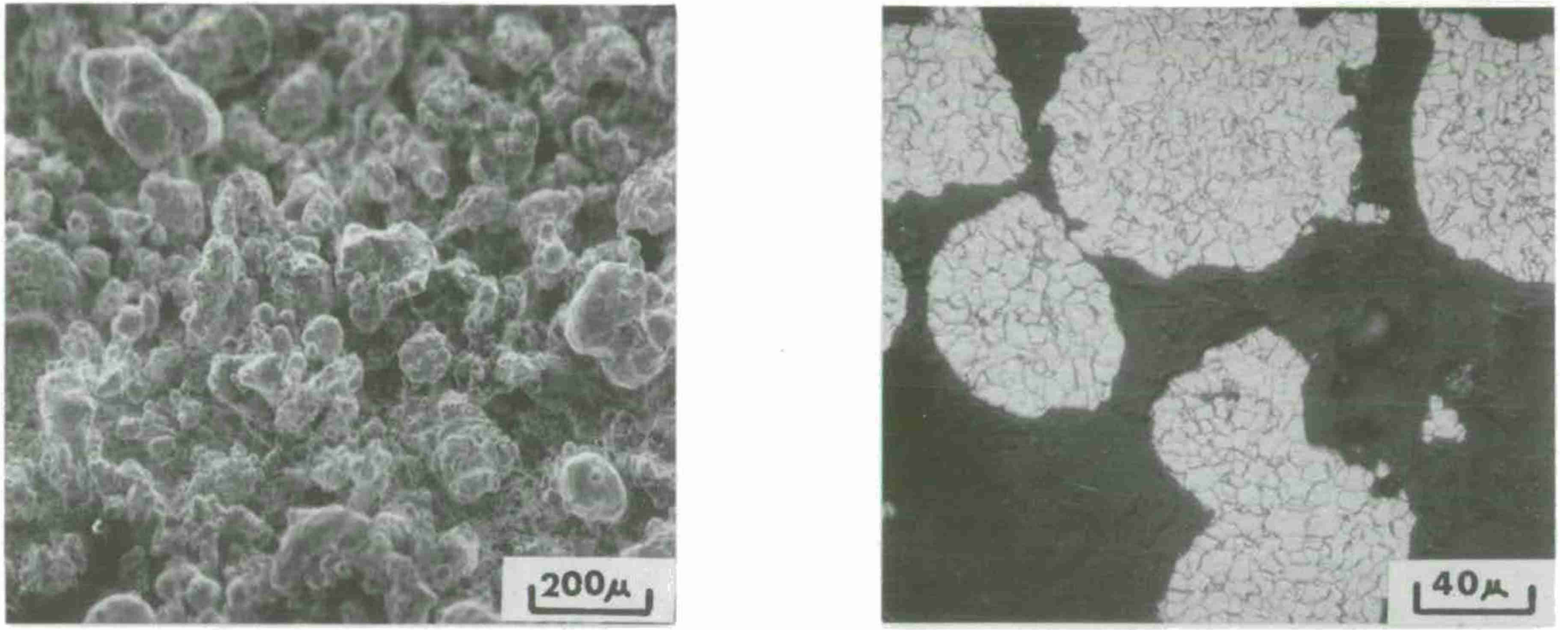


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**FIG. 1 PARTICLE SHAPE AND MICROSTRUCTURE OF 4600M POWDER**

Rectangular bars 3-1/2" x 3/4" x 1" were compacted with a graphite spray die wall lubricant to 80% of theoretical density. The preforms were batch sintered in a hydrogen-methane atmosphere followed directly by forging in a preheated die (350-400°F). The sintered preforms were forged by a "minimum deformation" process* resulting in approximately 5% lateral deformation during the forging operation. Final density, decarb depth, surface hardness, and oxygen content in the resulting forgings were used to evaluate the respective sintering parameters.

## Results and Discussion

The effects of various sintering parameters on the final forging P/M steel properties were studied. Parameters such as flow rate of hydrogen, flow rate of methane, sintering time, and sintering temperature were investigated. Since previous investigations have shown that oxygen content and final density in the forgings strongly affected impact and ductility properties, these criteria were used in evaluating the sintering parameters studied.

The residual oxide content as a function of hydrogen flow rate in the final forgings is shown in Figure 2. Various hydrogen flow rates ranging from 6 SCFH to 30 SCFH were used. The preforms were sintered at 2200°F for 40 minutes in a hydrogen-1% methane atmosphere. The methane addition prevents extensive decarburization during sintering. As noted from the resultant graph, the oxide content in the forgings decreases dramatically with increasing hydrogen flow rate. Increasing the flow rate from 6 SCFH to 30 SCFH reduces the oxide content by one-half. To meet military specification (MIL-F-45961) requirements for oxide content (<300ppm) in P/M steel forgings, a minimum hydrogen flow rate of 12 SCFH is necessary.

Decarb depth and surface hardness as a function of methane concentration are shown in Figures 3 and 4, respectively. The amount of decarburization during sintering decreases with increasing methane concentration. Decarb depths on the surface of the forgings range from 0.09 inch down to 0.001 inch, depending on the concentration of methane gas. Correspondingly, an increase in surface hardness is apparent with increasing methane concentration. To meet military specification requirements for surface hardness (Rc 30-33) and decarb depth (<0.001 inch) 5 percent by volume of methane is necessary in the flowing gases.

Various sintering times at 2200°F and 18 SCFH hydrogen flow rate were used to determine the optimum preform sintering time necessary for P/M steel forgings. The final densities of the forgings in relation to the various sintering times utilized are shown in Table 3. Sintering times in increments of 10 minutes (ranging from 10 to 60 minutes) were investigated. Sintering times of 30 minutes or longer are necessary to obtain P/M steel forgings with near-theoretical density (99.5 + percent). The effect of sintering time on the oxide content in the final forgings is shown in Figure 5. A dramatic decrease in oxide content is seen as the sintering time varies from 10 to 40 minutes. Sintering times greater than 40 minutes only gradually decrease the oxide content in the forgings.

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* Process developed by TRW under prior Government contract.

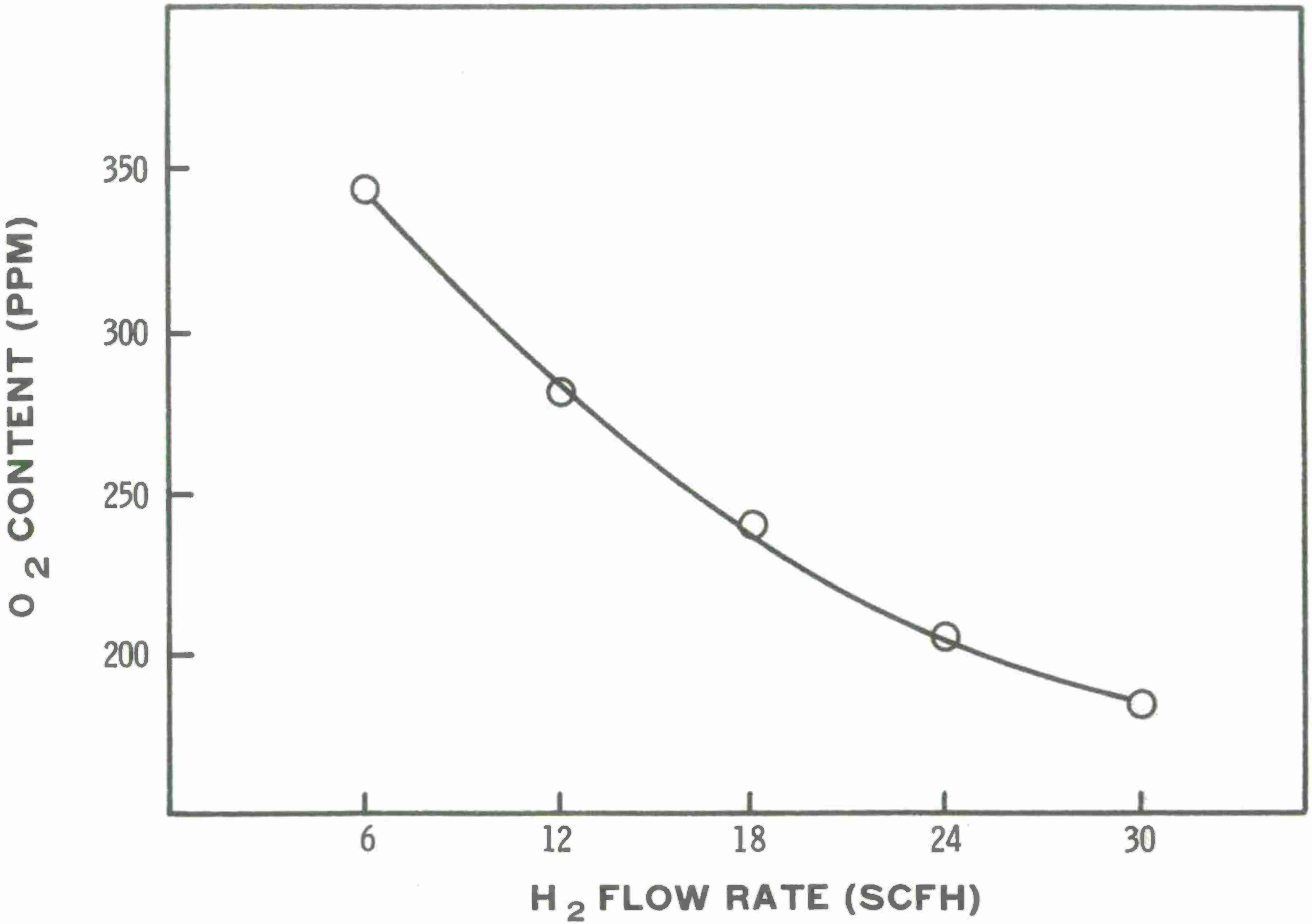



FIG. 2 THE EFFECT OF HYDROGEN FLOW RATE ON REDUCTION OF OXIDES

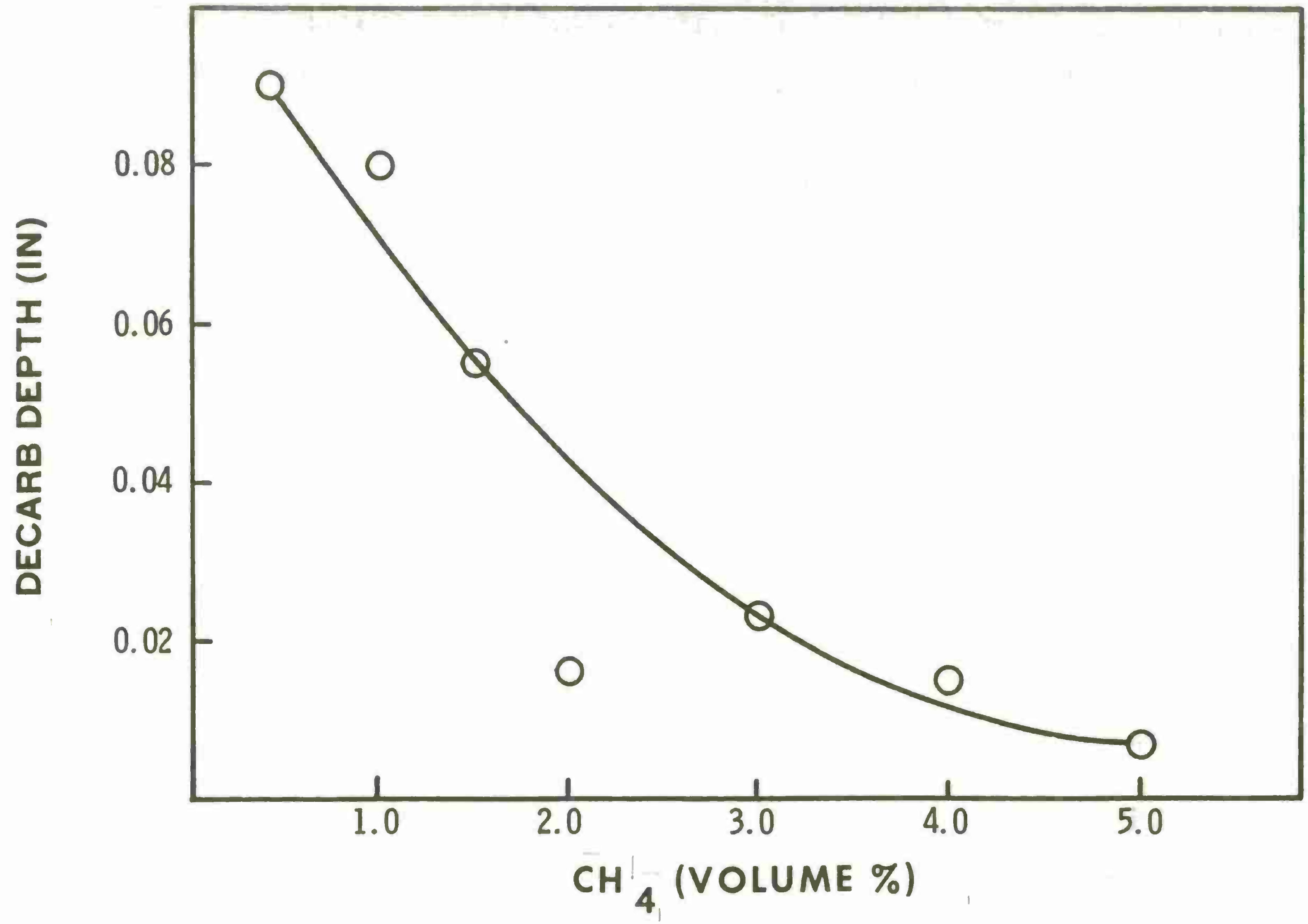



**FIG. 3 THE EFFECT OF METHANE CONCENTRATION IN SINTERING ATMOSPHERE ON SURFACE DECARBURIZATION**

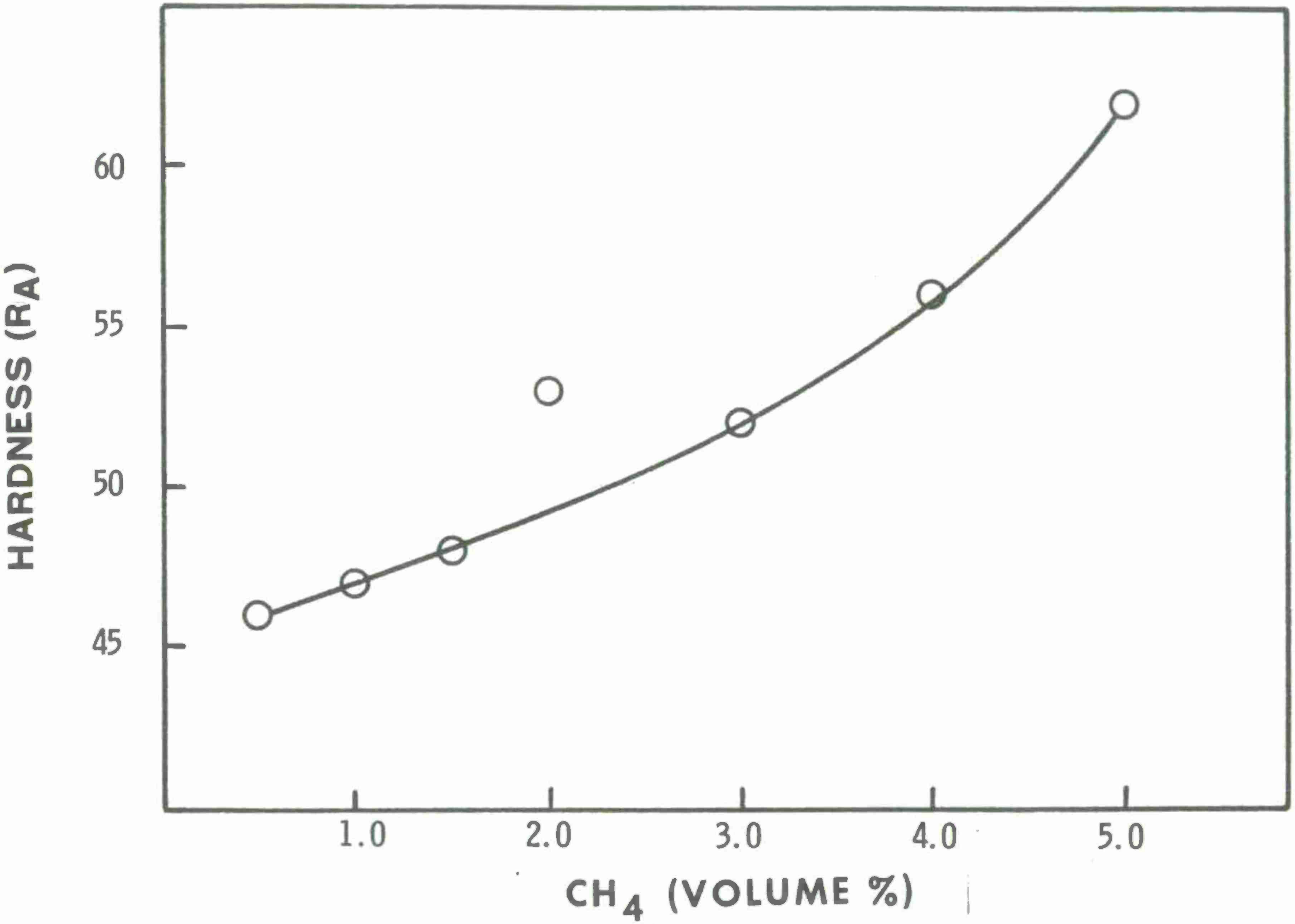



FIG. 4 THE EFFECT OF METHANE CONCENTRATION IN SINTERING ATMOSPHERE ON SURFACE HARDNESS

TABLE 3

SINTERING TIME - FORGED DENSITY OF 4600 PREALLOYED POWDER

| Sintering Time (min) | Density ($g/cm^3$) | Density (%) |
|---|---|---|
| 10 | 7.78 | 99.0 |
| 20 | 7.78 | 99.0 |
| 30 | 7.83 | 99.6 |
| 40 | 7.83 | 99.6 |
| 50 | 7.83 | 99.6 |
| 60 | 7.83 | 99.6 |

TABLE 4

SINTERING TEMPERATURE - FORGED DENSITY OF 4600 PREALLOYED POWDER

| Sintering Temperature (°F) | Density ($g/cm^3$) | Density (%) |
|---|---|---|
| 1900 | 7.80 | 99.2 |
| 2000 | 7.78 | 99.0 |
| 2100 | 7.81 | 99.4 |
| 2200 | 7.83 | 99.6 |

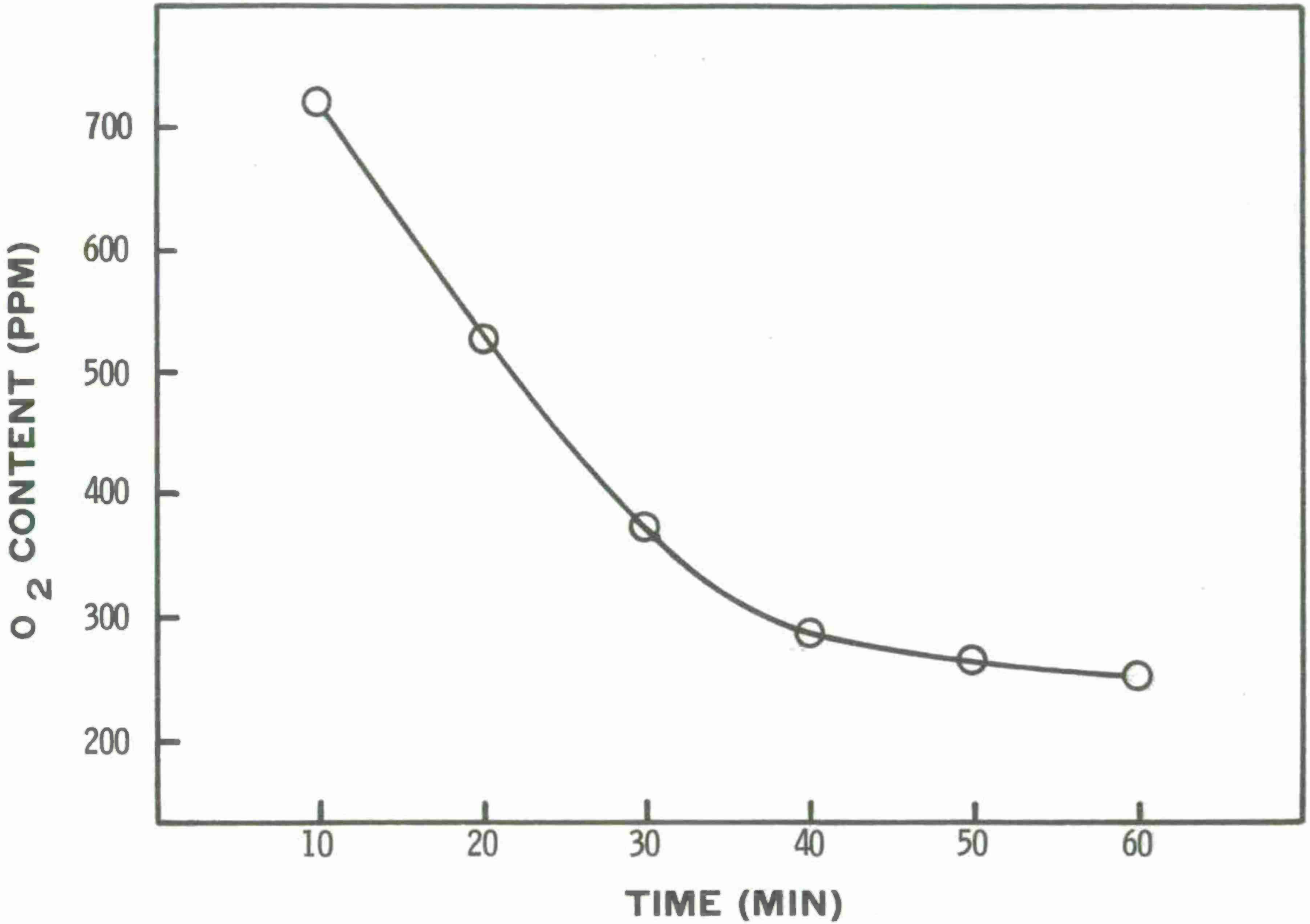



FIG. 5 SINTERING TIME EFFECT ON OXYGEN CONTENT IN P/M STEEL FORGINGS

Four preform sintering temperatures (1900, 2000, 2100, and 2200°F) were investigated to determine the effect of sintering temperature on P/M forged properties. A sintering time of 40 minutes and a hydrogen flow rate of 18 SCFH were used. The relationship between final forged density and preform sintering temperature is shown in Table 4. An increase in sintering temperature (1900 to 2200°F) results in a slight increase in the forged density (99.2 to 99.6%). Sintering temperature has a significant effect on the oxide content in the resultant forging, as shown in Figure 6. Oxide content dramatically drops as the sintering temperature is increased from 1950 to 2200°F. The oxide content of the forging is approximately 1400 ppm at 1900°F, whereas, at 2200°F the oxide content is below 200 ppm.

## POWDER DISTRIBUTION STUDIES

### Procedure

Commercially available prealloyed 4600 powder was separated into individual fractions and blended to form various unimodal and bimodal distributions. The distributions were formulated from statistical models described by the binomial distribution. The equations used were:

$$\frac{n!}{x!(n-x)!} p^{x} (1-p)^{n-x} \qquad \text{for } x = 0, 1, \ldots\ldots n \qquad (1)$$

$$\frac{n!}{x!(n-x)!} (0.5)^{n} \qquad \text{for normal distribution} \qquad (2)$$

where n is the number of data points which establishes the distribution curve and p is the fraction of large particles in the distribution. At p=0.5, the distribution will have an even, normal distribution of large and small particles.

Five values of P(0.2, 0.4, 0.5, 0.6, and 0.8) were used for the unimodal distributions. The five unimodal distributions obtained are shown in Figure 7. At p=0.2 and 0.8 the distributions are skewed towards smaller and larger size particles, respectively. The two distributions in the middle, p=0.4 and p=0.6, are distributions slightly skewed from the normal.

The type of bimodal distributions used was designed by incorporating two values of p from the unimodal curves. After careful selection of a variety of bimodal distributions, four bimodal distributions were used that would cover the range of interest (see Figure 8). A combination of p=0.1 and p=0.6 forms a bimodal distribution predominantly of smaller size particles. The distribution p=0.1 + 0.9 is a bimodal distribution of equal weights of very large and very small size particles. A combination of p=0.2 and p=0.8 forms a normal bimodal distribution. The last distribution, p= 0.4 + 0.9, is a bimodal distribution predominantly of large size particles.

The powder blends were characterized according to size and size distributions, apparent density, and flow rate. Individual particles were evaluated by scanning electron microscopy and metallography. The powder blends were mixed with 0.4% flake carbon, compacted to 80% theoretical density, sintered at 2200°F for 40 minutes, and forged in a warm closed die to

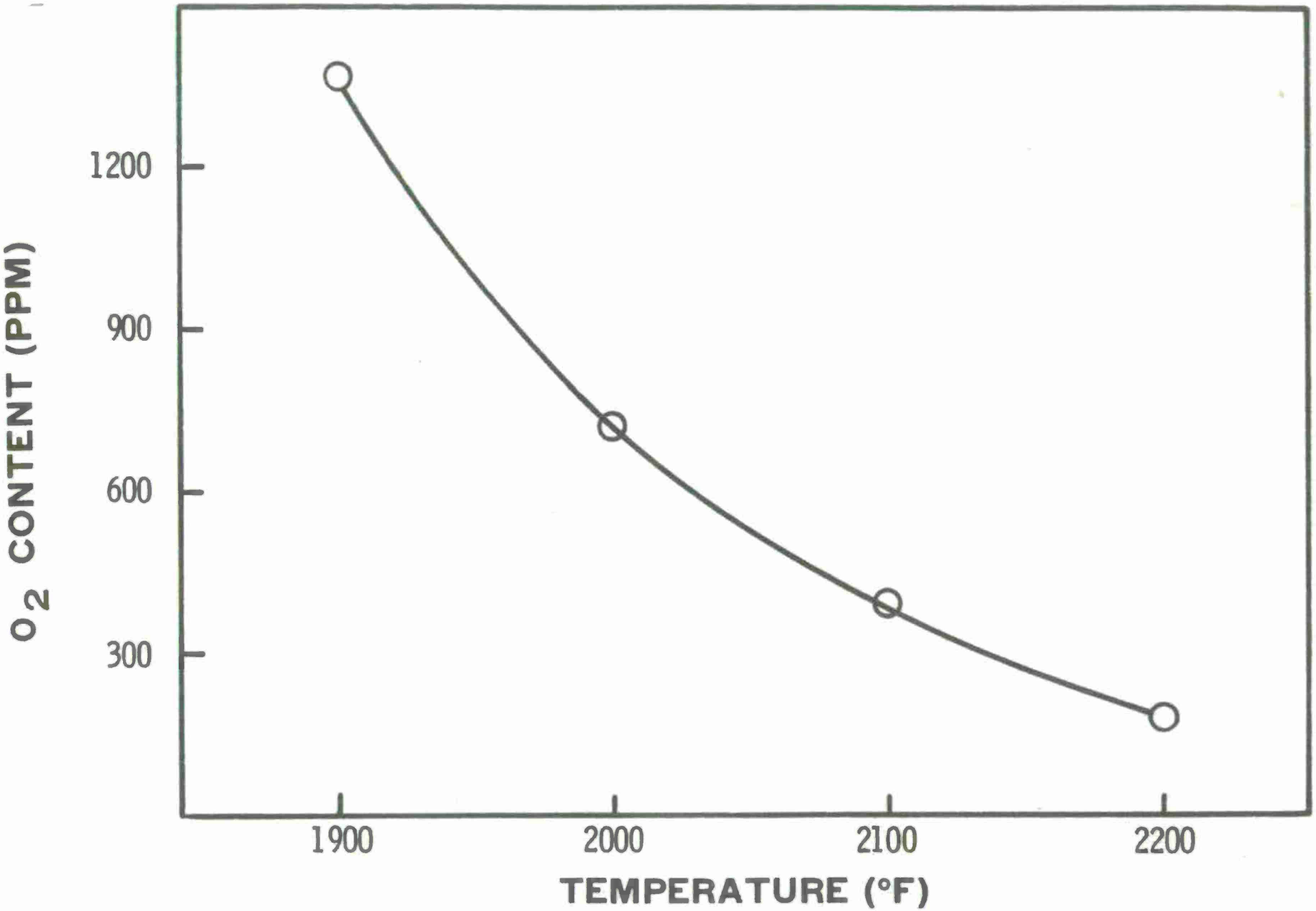



**FIG. 6 SINTERING TEMPERATURE EFFECT ON OXYGEN CONTENT IN P/M STEEL FORGINGS**

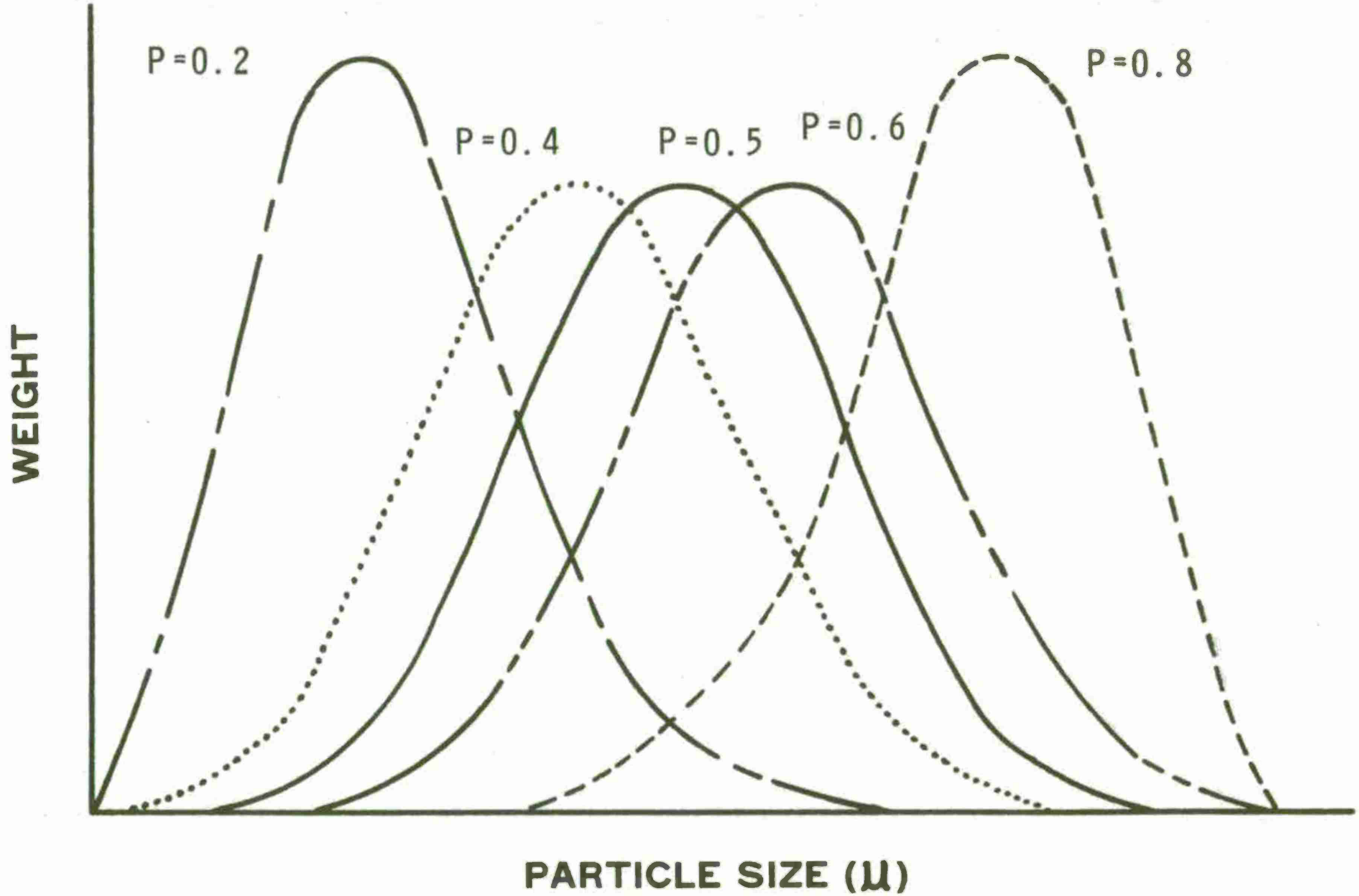



FIG. 7 UNIMODAL DISTRIBUTIONS

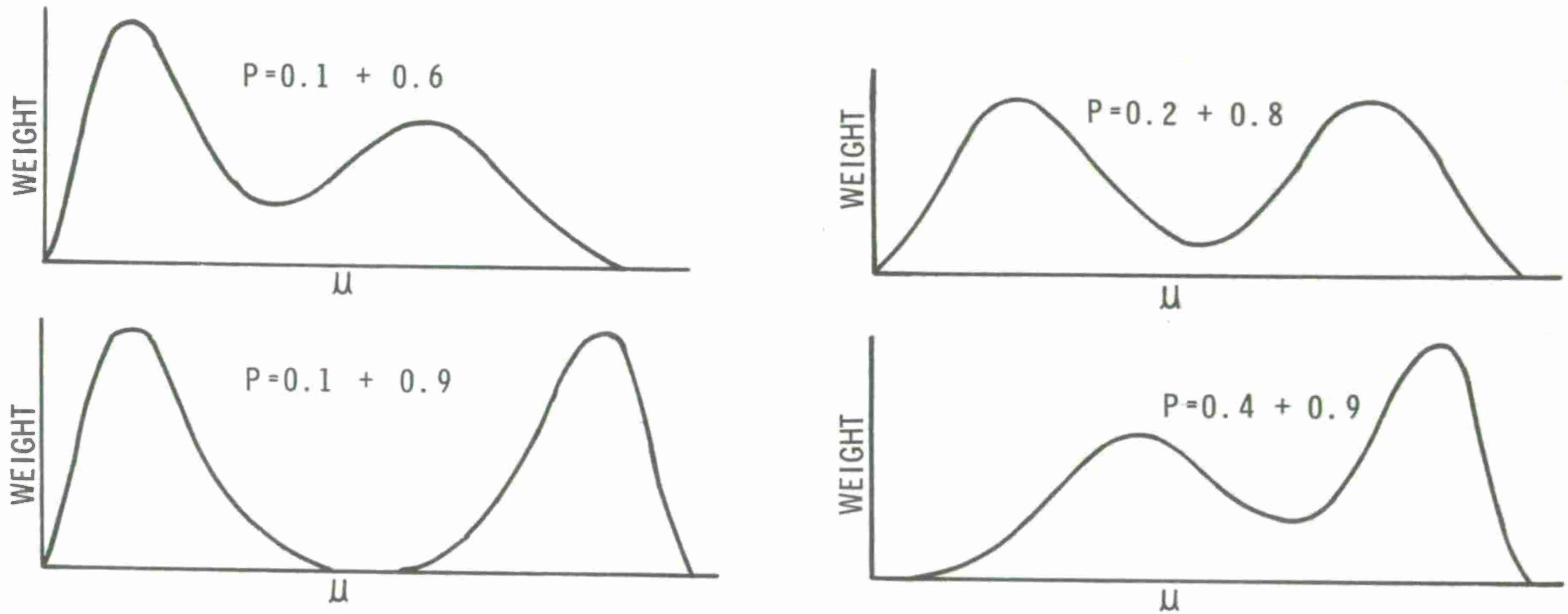



FIG. 8 BIMODAL DISTRIBUTIONS

produce fully dense forgings. The forgings were tested for density, grain size, decarburization, elongation, yield and tensile strengths, carbon and oxygen contents, and Charpy impact energy.

## Results and Discussion

The powder characteristics for the unimodal distributions are shown in Table 5. Apparent densities of the unimodal distributions were found to decrease with increasing particle size. Correspondingly, the flow rates were found to increase with the particle size of the distribution. Table 6 on bimodal distributions also shows similar relationships with particle size and the powder characteristics of apparent density and flow rate.

Powder characteristic differences were strongly influenced by the shapes and sizes of the individual particles in the distributions. The shapes and sizes of powder particles in two distributions are shown in Figure 9. The smaller size particles (represented by a p=0.2 distribution) are relatively smooth and essentially spheroidal. The larger size particles (represented by a p=0.8 distribution) have a variety of shapes and are characterized by a "popcorny" nature. These characteristic differences in shapes also influence the green strengths of the compacted preforms. As expected, preforms compacted from large size particle distributions have greater green strengths than preforms from the smaller size particle distributions. For example, at $6.3 g/cm^3$ the green strength for the p=0.2 distribution is 1530 psi, compared to 1810 psi for the p=0.8 distribution. This increase in green strength occurs because the "popcorn-like" shape of the large size particles can achieve better interlocking among the individual particles than the relative smooth shape prevalent in the smaller size particles.

The objective at the outset on the study of powder distributions was to determine what effect (if any) powder distributions had on final forged properties. Table 7 shows the mechanical and physical properties of the final forgings from the various unimodal powder distributions. The densities, tensile strengths, and yield strengths have little variation from one powder distribution to another. However, the ductility properties, impact strength, and oxygen content are influenced by the particle size distributions. The relationship between particle size and oxygen content in the final forging is shown in Figure 10. The oxygen content varies linearly with the particle size. An increase in particle size shows a corresponding increase in the oxygen content of the P/M forging. Consequently, increasing the particle size decreases the impact energy as shown in Figure 11. The log-log relationship established in Figure 11 is described by the empirical equation

$$W=58\mu^{-0.22} \qquad (3)$$

where W is the impact energy and $\mu$ is the particle size. The effect of particle size on ductility properties is shown in Figure 12. A decrease in particle size increases the elongation and reduction in area in the forgings. The ductility properties are strongly influenced by the particle size up to a critical value ($\sim 80\mu$). Particle sizes smaller than $80\mu$ only slightly increase the ductility properties.

## TABLE 5

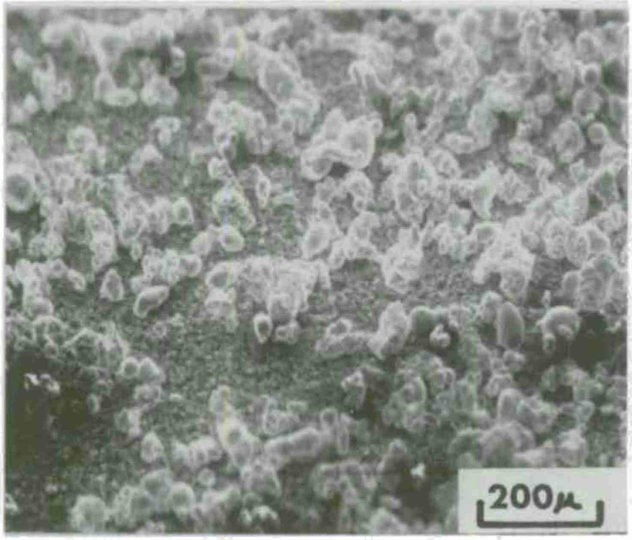
## POWDER CHARACTERISTICS OF UNIMODAL DISTRIBUTIONS

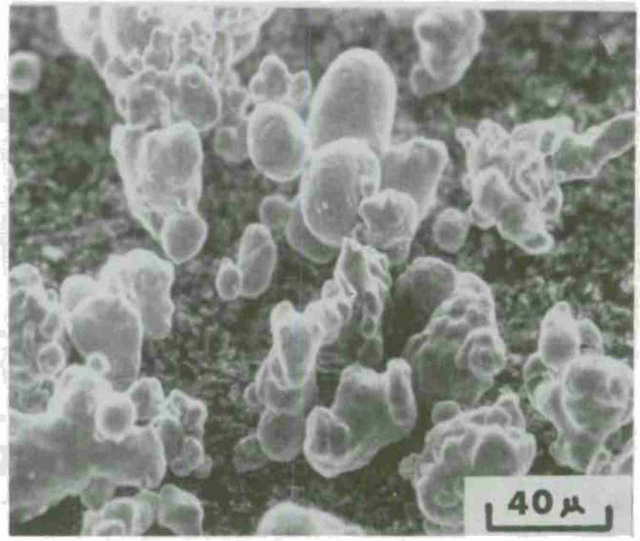
| | Sieve Analysis, wt % | | | | |
|---|---|---|---|---|---|
| U.S. Screen Size | P=0.2 | P=0.4 | P=0.5 | P=0.6 | P=0.8 |
| -100 (149μ) | ---- | 0.1 | 0.3 | 2.5 | 20.3 |
| -100 + 140 | 0.3 | 8.7 | 23.6 | 45.3 | 70.7 |
| -140 + 200 | 5.9 | 39.5 | 49.1 | 42.3 | 8.6 |
| -200 + 230 | 10.6 | 18.2 | 12.8 | 6.0 | 0.4 |
| -230 + 325 | 31.6 | 22.9 | 11.6 | 3.5 | ---- |
| -325 (44μ) | 51.6 | 10.6 | 2.6 | 0.4 | ---- |
| Apparent density ($g/cm^3$) | 2.88 | 2.82 | 2.76 | 2.76 | 2.77 |
| Flow rate (sec) | 28.3 | 29.2 | 30.7 | 31.9 | 33.2 |

TABLE 6

POWDER CHARACTERISTICS OF BIMODAL DISTRIBUTIONS

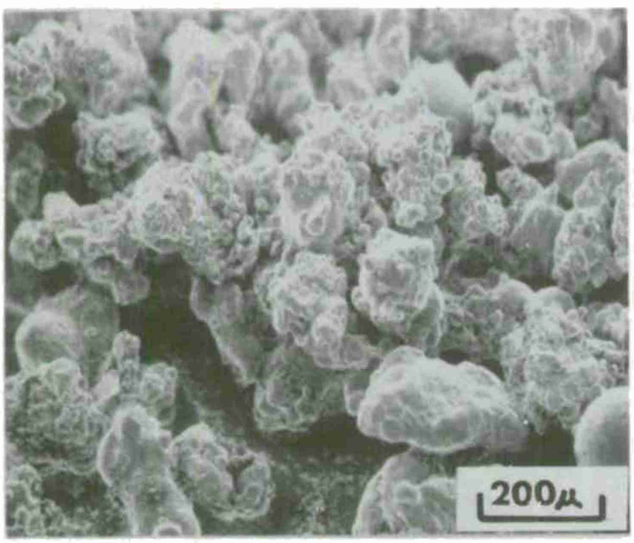
| U.S. Screen Size | Sieve Analysis, wt % P=0.1+0.6 | P=0.1+0.9 | P=0.2+0.8 | P=0.4+0.9 |
|---|---|---|---|---|
| -100(149μ) | 1.2 | 26.3 | 10.2 | 25.2 |
| -100 +140 | 23.0 | 23.9 | 35.6 | 29.3 |
| -140 +200 | 21.2 | 0.3 | 7.8 | 20.1 |
| -200 +230 | 4.0 | 1.0 | 4.9 | 9.1 |
| -230 +325 | 8.8 | 6.6 | 15.7 | 11.3 |
| -325(44μ) | 41.9 | 41.8 | 25.7 | 4.9 |
| Apparent density ($g/cm^3$) | 2.98 | 3.13 | 3.01 | 2.92 |
| Flow rate (sec) | 27.2 | 26.7 | 27.6 | 29.7 |

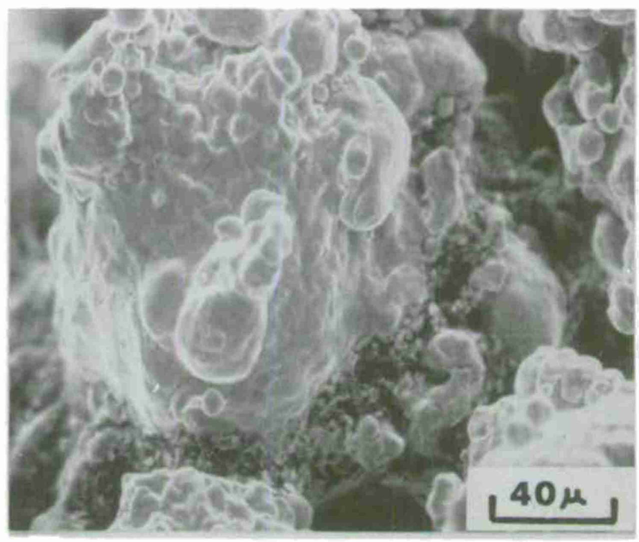



P=0.2

P=0.2




P=0.8

P=0.8

**FIG. 9 PARTICLE SHAPES AND SIZES IN POWDER DISTRIBUTIONS**

## TABLE 7

## PROPERTIES OF FORGINGS FROM UNIMODAL DISTRIBUTIONS

| | Distribution | | | | |
|---|---|---|---|---|---|
| Properties | 0.2 | 0.4 | 0.5 | 0.6 | 0.8 |
| Density (%) | 99.5 | 99.6 | 99.5 | 99.5 | 99.6 |
| Tensile (ksi) | 138 | 140 | 138 | 139 | 139 |
| Yield (ksi) | 132 | 133 | 131 | 132 | 131 |
| Elongation (%) | 15.1 | 14.4 | 13.3 | 12.3 | 13.3 |
| R/A (%) | 44.8 | 44.6 | 42.7 | 38.0 | 38.6 |
| Impact (ft-lbs) | 25.5 | 22.5 | 22.0 | 21.0 | 20.0 |
| $O_2$ (ppm) | 158 | 185 | 203 | 241 | 246 |

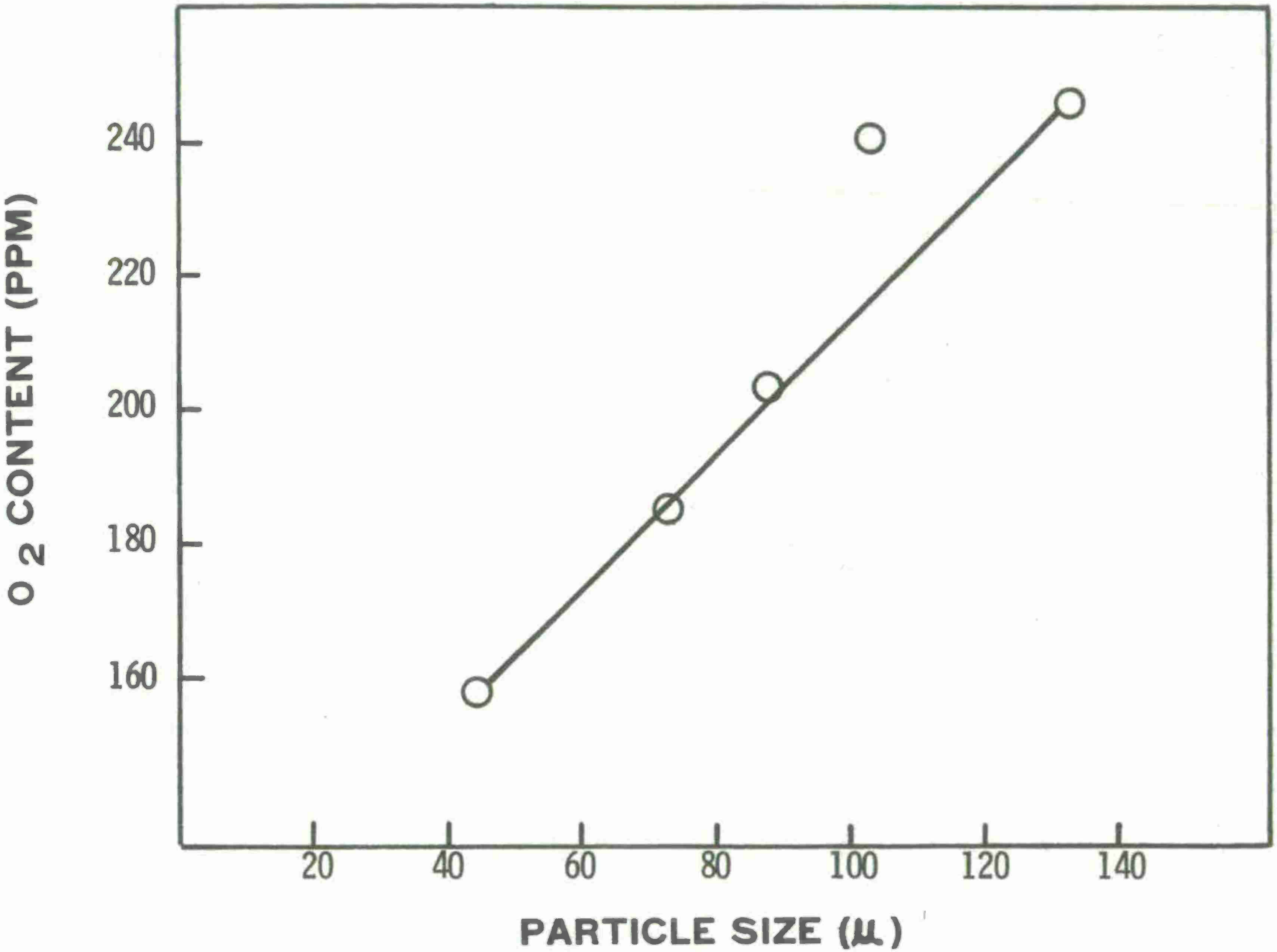



FIG. 10 OXYGEN CONTENT OF UNIMODAL FORGINGS AS A FUNCTION OF PARTICLE SIZE

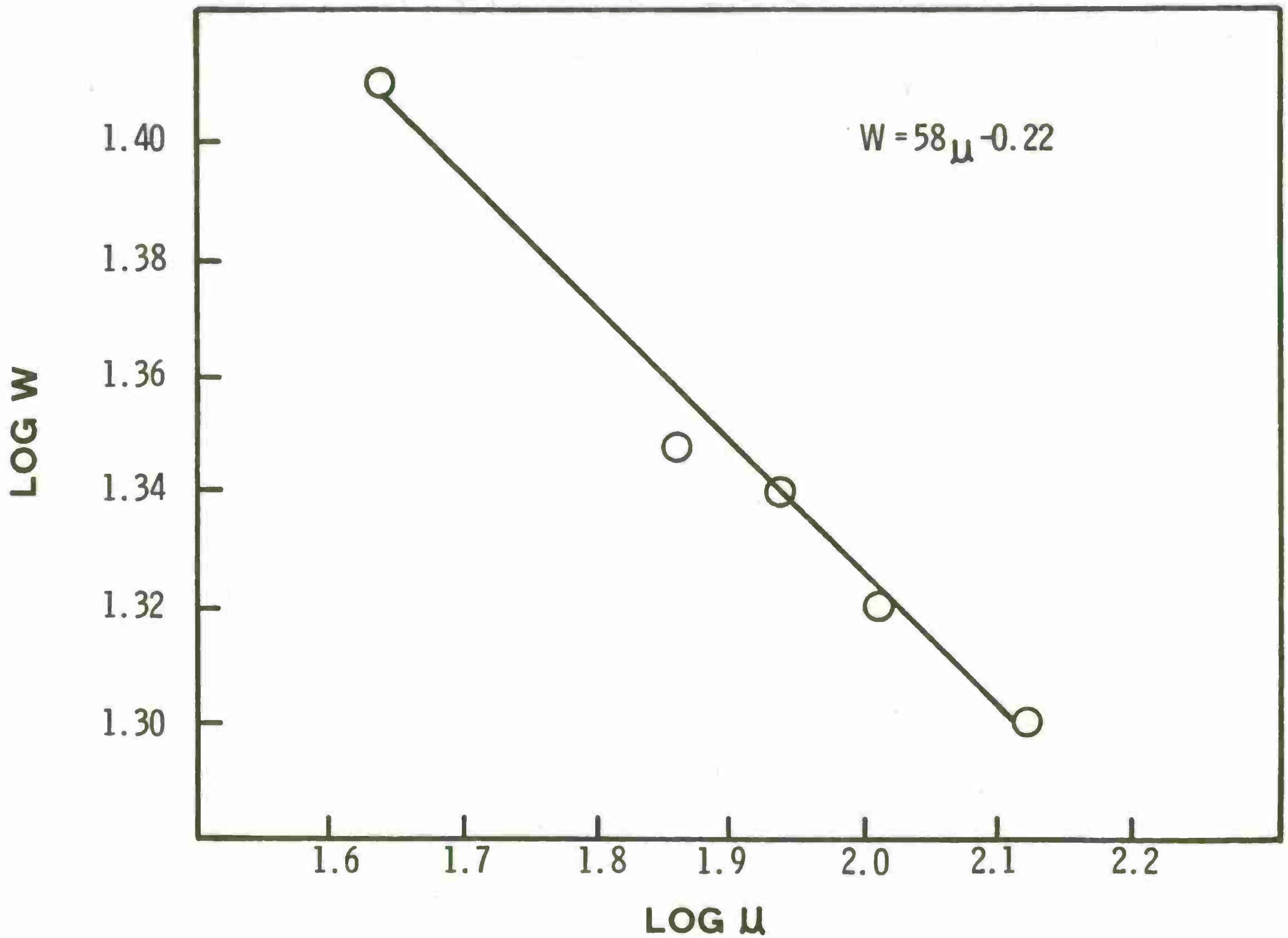



FIG. 11 IMPACT ENERGY OF UNIMODAL FORGINGS AS A FUNCTION OF PARTICLE SIZE

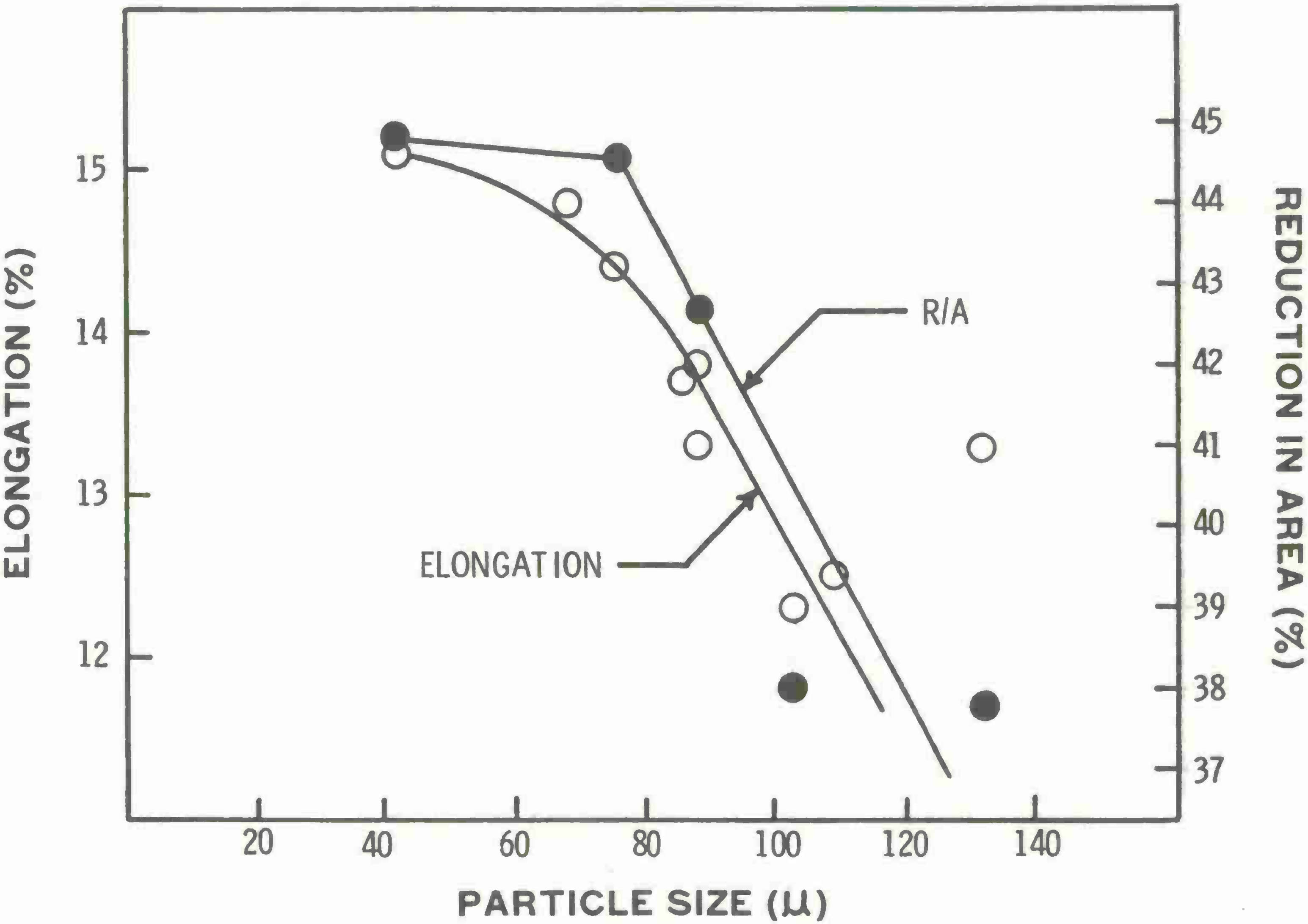
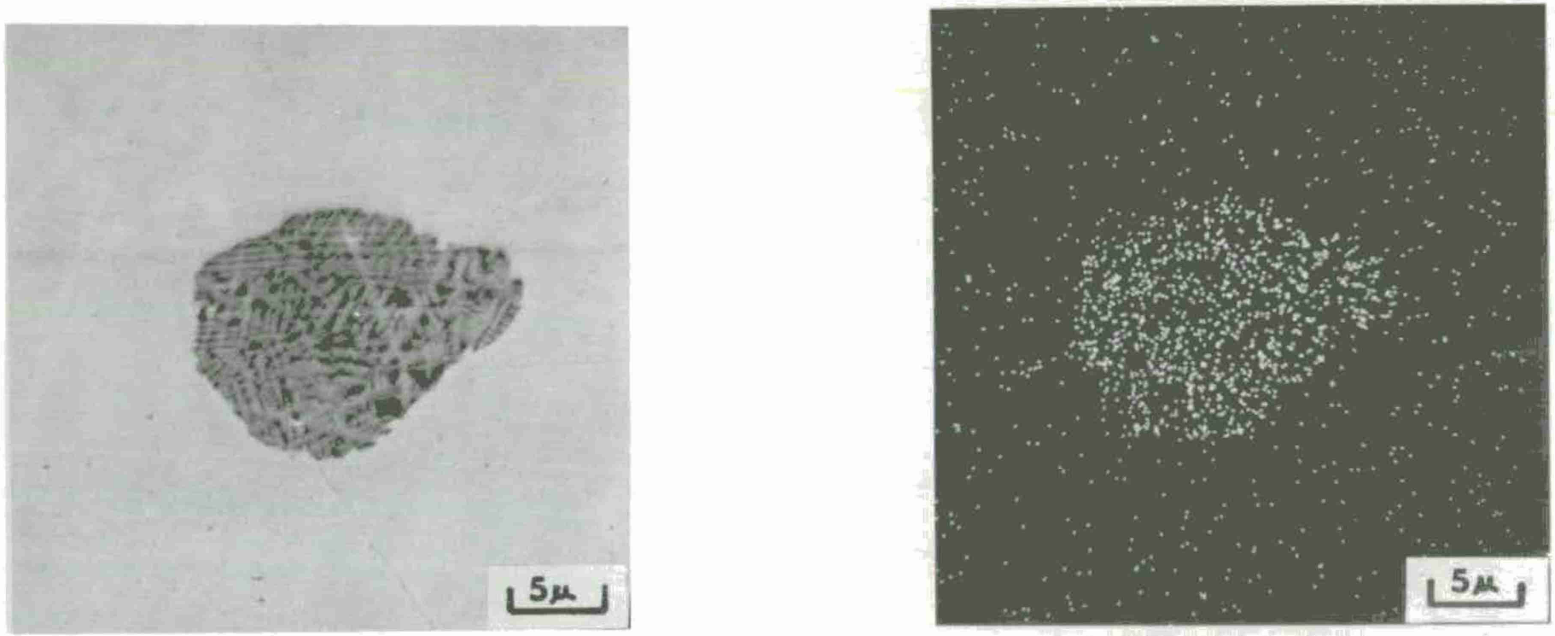



FIG. 12 DUCTILITY PROPERTIES OF UNIMODAL FORGINGS AS A FUNCTION OF PARTICLE SIZE

## TABLE 8

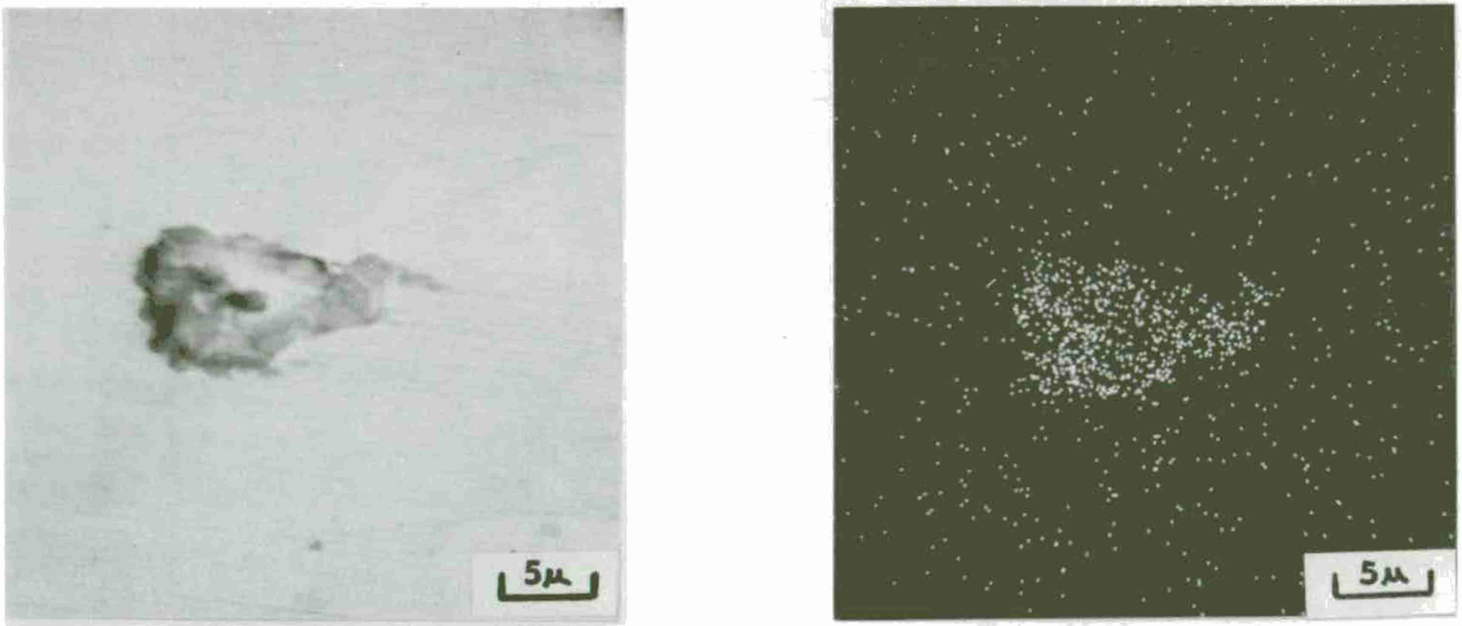
## PROPERTIES OF FORGINGS FROM BIMODAL DISTRIBUTIONS

| Properties | Distribution 0.1 + 0.6 | 0.1 + 0.9 | 0.2 + 0.8 | 0.4 + 0.9 |
|---|---|---|---|---|
| Density (%) | 99.8 | 99.5 | 99.4 | 99.5 |
| Tensile (ksi) | 136 | 140 | 138 | 136 |
| Yield (ksi) | 128 | 134 | 133 | 129 |
| Elongation (%) | 14.8 | 13.8 | 13.7 | 12.5 |
| R/A (%) | 49.0 | 44.0 | 44.8 | 44.0 |
| Impact (ft-lbs) | 23.0 | 21.5 | 22.5 | 21.0 |
| $O_2$ (ppm) | 183 | 191 | 197 | 200 |




A B

FIG. 13 SCANNING ELECTRON (A) AND MICROPROBE (B) PHOTOGRAPHS OF MANGANESE OXIDE INCLUSION IN 4600 M POWDER PARTICLE




A B

FIG. 14 SCANNING ELECTRON (A) AND MICROPROBE (B) PHOTOGRAPHS OF MANGANESE OXIDE INCLUSION IN P/M STEEL FORGING

Mechanical and physical properties for bimodal distributions, as shown in Table 8, show results similar to the unimodal distributions. Again, particle size influences the ductility properties, impact energy, and oxygen content in the P/M forgings. The P/M forging from the p=0.1 + 0.6 distribution has the highest impact energy, percent elongation, and percent reduction in area as well as the lowest oxygen content.

The differences in mechanical and physical properties for the various powder distributions can be explained by a closer examination of forging and powder particle cross sections. Examination of powder particle cross sections by SEM reveal inclusions in some of the large size particles (see Figure 13A). The characteristic dendritic structure of the inclusions is typical of manganese oxide. Positive identification of a concentration of manganese is shown by microprobe analysis (see Figure 13B). Further analysis of sectioned forgings by SEM shows the manganese oxide inclusion still present (see Figure 14). The manganese oxide inclusions are more prevalent in forgings forged from large size particles than from small size particles. Thus, complete reduction of the manganese oxide inclusion does not occur under the prevalent sintering conditions and could be the reason for the property differences noted in the powder distribution forgings.

## CONCLUSIONS

Selection of proper sintering conditions is paramount to obtain optimum physical and mechanical properties in P/M steel forgings. The oxide content and final density in P/M steel forgings are strongly dependent on sintering temperature, sintering time, and flow rate of reducing gas (hydrogen). Increasing the sintering temperature in the range of 1900 to 2200°F decreases the final oxide content sevenfold. Correspondingly, increasing the flow rate of a reducing gas significantly reduces the final oxide content. This effect is due to the lowering of the resultant dew point in the sintering furnace. A normal sintering time of 40 to 60 minutes is sufficient for P/M steel preforms before forging. Longer sintering times only gradually decrease the resultant oxide content. To prevent decarburization during sintering, careful control of the flow rate of the carburizing gas (methane) is necessary. The decarb depth and surface hardness of the final forging is significantly affected by the carburizing gas concentration. Five-percent methane in the flowing gas is necessary to prevent decarb depths of more than 0.001 inch.

The size distribution of the powder particles influences the resultant density, oxygen content, impact strength, and ductility properties in P/M steel forgings. The property differences relate to the incomplete reduction of manganese oxide inclusions prevalent in many of the large size powder particles. The powder distribution study showed a gradual decrease in the mechanical properties of the forgings with increasing powder distribution size. Whether this decrease is significant or not, depends entirely on the specification requirements for the particular P/M steel forging application.

## FUTURE WORK

Fundamental studies on the deformation and fracture behavior of sintered 46XX powder preforms at hot working temperatures will be initiated. The deformation and fracture behavior will be studied systematically under a range of stress and strain states by means of upset tests. The analytical stress-strain relationships established will be utilized to determine the design criteria for sintered steel preforms for forging. Design criteria to be established are: attainment of full density and a sound metallurgical structure, and prevention of fracture during forging.

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The effects of sintering parameters and particle size distributions on the properties of steel P/M forgings were investigated. Sintering parameters studied were hydrogen flow rate, methane flow rate, and sintering time and temperature. Various unimodal and bimodal particle size distributions designed from statistically determined powder blends were investigated in the powder distribution study. Oxygen content and final density in the forgings strongly depend on the proper selection of the sintering parameters. Among the sintering parameters

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